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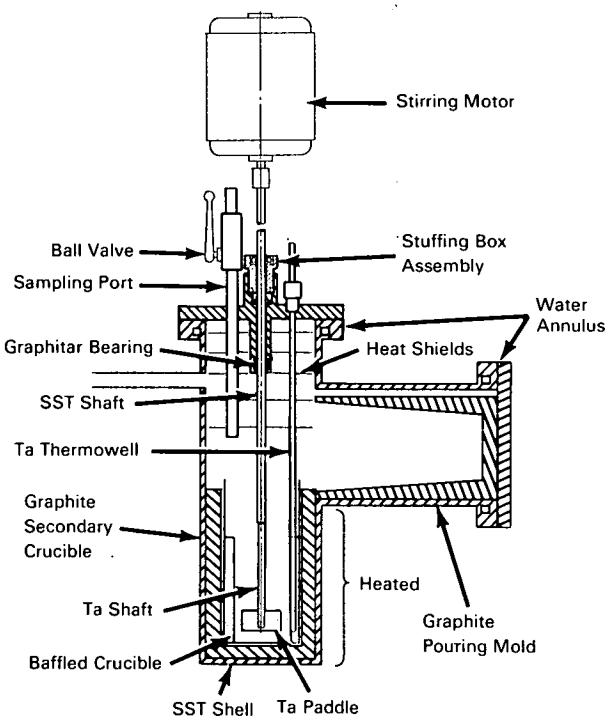
AEC-NASA TECH BRIEF

Preparation of Thorium by Magnesium–Zinc Reduction



THORIUM DIOXIDE REDUCTION APPARATUS

A technique for the preparation of thorium metal by magnesium–zinc reduction of thorium dioxide has been investigated on a laboratory scale. The extent of reduction was determined as a function of time, amount and composition of flux, temperature, agitation, particle size of the oxide, and magnesium concentration in the alloy. It was found that the use of a flux of the proper composition is essential to the success of the process.

The present method for commercial production of thorium metal involves metallothermic reduction of thorium tetrafluoride with calcium in the presence of zinc chloride. However, calcium is expensive as a reductant and the process requires a prior hydro-fluorination step. Using magnesium–zinc alloy for the reduction of thorium dioxide has potential economic

(continued overleaf)

advantages; relatively inexpensive reagents are used for the metal and flux phases, and metal of acceptable quality can be produced in good yield.

For the experiments, the molten salt fluxes were prepared from anhydrous, reagent-grade salts, except for magnesium chloride, which was obtained as a by-product from a process used for the production of zirconium. The thorium dioxide was a high-fired material that had been screened to –325 mesh. The zinc and magnesium were 99.99 and 99.95% pure, respectively. Preliminary reductions were performed in the open air.

Although complete reductions were achieved in air atmosphere, the rate and extent of reduction were improved by the use of an argon atmosphere. The apparatus shown in the figure was used for the argon atmosphere experiments. It consisted primarily of a resistance-heated, tilt-pour, stainless-steel furnace. The reductions were conducted in a tantalum crucible, equipped with four baffles to enhance mixing, and located inside a graphite secondary container. By tilting the entire furnace, the molten contents of the crucible could be poured into the mold. The furnace was also equipped with a thermocouple wall, a sampling port, and provisions for applying vacuum or an argon atmosphere.

Under optimum conditions, over 99% reduction of thorium dioxide was obtained in less than 4 hours, with product purities up to 99.7%. These optimum conditions were: 800°C, argon atmosphere, a magnesium conc. of 5 to 10 w/o in the zinc phase, a flux containing a large proportion of magnesium chloride and $\approx$ 10 m/o calcium or magnesium fluoride, and a thorium dioxide; flux weight ratio of about 0.22%.

In addition, four small-scale demonstration experiments were performed, in which thorium dioxide was reduced by zinc–magnesium alloy. The resulting liquid-metal solution was distilled to produce a thorium metal sponge, which was consolidated by arc-melting.

Notes:

1. Procedures and results have been published in "Preparation of Metals by Magnesium-Zinc Reduction—Part II. Reduction of Thorium Dioxide," ANL-7058, June 1965, by A. V. Hariharan, J. B. Knighton, and R. K. Steunenberg of Argonne National Laboratory. This report is available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151; price: $3.00 (microfiche copies, $0.65), Reference: 69-10079. Additional information is also contained in Patent No. 3,164,462 (January 5, 1965) "Preparation of Thorium Metal from the Oxide," which is available from the U. S. Patent Office at $0.50 each.
2. Thorium metal is used in electronics industries for its thermionic emission, low work function, and action as a "getter." Since this information may help to improve production processes of thorium, it should be of interest to persons in the electronic industries.
3. Inquiries concerning this innovation may be directed to:

   Office of Industrial Cooperation
   Argonne National Laboratory
   9700 South Cass Avenue
   Argonne, Illinois 60439
   Reference: B69-10079
   Source: A. V. Hariharan, J. B. Knighton, and R. K. Steunenberg (ARG-10245)

Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

Mr. George H. Lee, Chief
Chicago Patent Group
U. S. Atomic Energy Commission
Chicago Operations Office
9800 South Cass Avenue
Argonne, Illinois 60439